

HAZARDOUS WASTE PROGRAM:

ANALYTICAL DATA

DELIVERABLE

REQUIREMENTS

FOR RCRA CLOSURES, RISK ASSESSMENTS, SITE ASSESSMENTS, AND REMEDIATION PROJECTS

Chemistry Section
Office of Land Quality
Indiana Department of Environmental Management

ANALYTICAL DATA DELIVERABLE REQUIREMENTS **FOR** **RCRA CLOSURES, RISK ASSESSMENTS,** **SITE ASSESSMENTS, AND REMEDIATION PROJECTS**

INTRODUCTION

The purpose of this document is to provide guidance to Resource Conservation and Recovery Act (RCRA) facilities submitting analytical data in support of risk assessments, closures, and other remediation-related projects to the Indiana Department of Environmental Management (IDEM). This includes data submitted in conjunction with voluntary remedial actions for purposes of RCRA closure, RCRA corrective action, and other RCRA-related remediation, whether data is submitted through the Voluntary Remediation Program (VRP) (in applications, work plans, reports, etc.) or submitted directly to hazardous waste staff in the Office of Land Quality (OLQ). **Facilities are responsible for notifying the environmental consultants managing their remedial activities and the laboratories analyzing their samples of the required data deliverables *prior* to sampling and analysis activities. This will ensure that appropriate control measures are taken and that documentation of such measures is readily available.**

This document is not intended to address data deliverable requirements for RCRA Part B Permit applications or permit-related waste characterizations. (For data deliverable requirements for permits, see draft document "Analytical Data Deliverable Requirements for RCRA Permits: A Guidance Document," May 1995. This document will be finalized after further revision but provides the basic requirements for permit-related analysis.)

For general guidance in preparing risk assessments, please refer to *Risk Assessment Guidance for Superfund (RAGS)* developed by the United States Environmental Protection Agency (USEPA).¹ For additional information, contact the Chemistry Section, at (317)232-8929.

¹ For evaluation of human health risk during the investigation phase, see: Environmental Protection Agency, Office of Emergency and Remedial Response, Toxics Integration Branch, *Risk Assessment Guidance for Superfund, Volume I, Human Health Evaluation Manual*:

- (Part A), *Interim Final*, EPA/540/1-89/002, December 1989;
- (Part B, *Development of Risk-based Preliminary Remediation Goals*), OSWER Directive 9285.7-01B, December 1991.

For evaluation of ecological risk during the investigation phase, see: Environmental Protection Agency, Office of Emergency and Remedial Response, Toxics Integration Branch, *Risk Assessment Guidance for Superfund, Volume II, Environmental Evaluation Manual: Interim Final*, EPA/540/1-89/001, March 1989; and

- USEPA Region 5, Waste Management Division, Office of RCRA, "Ecological Risk Assessment Guidance for RCRA Corrective Action: Region 5: Interim Draft," October 1994.

REMEDIAL OBJECTIVE AND DATA QUALITY OBJECTIVE

The DQO. The quality level required for analytical data obtained in support of an environmental project is known as the Data Quality Objective (DQO). The DQO for a remedial action is dependent on the objective, or goal, of the particular project. For example, the project goal might be to clean up site contamination to background concentrations. Or, the project objective might be to perform a risk assessment to demonstrate that remedial action is not required because contamination levels at the site do not pose a threat to human health or the environment.

The DQO concept and the process for determining project DQOs were developed by USEPA to assist in the collection of data important to decision making in environmental projects. In its DQO guidance document, the USEPA states:

The process allows decision makers to define their data requirements and acceptable levels of decision errors^[*] during planning, before any data are collected. Application of the DQO Process should result in data collection designs that will yield results of appropriate quality for defensible decision making.

[*] Decision errors occur when variability or bias in data mislead the decision maker into choosing an incorrect course of action. . . .²

The USEPA (EPA) defines DQOs and the DQO development process as follows:

What is the DQO Process? The DQO Process is a series of planning steps based on the Scientific Method that is designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application. The steps of the DQO Process are illustrated in Figure 1 [below].

What are DQOs? DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO Process that:

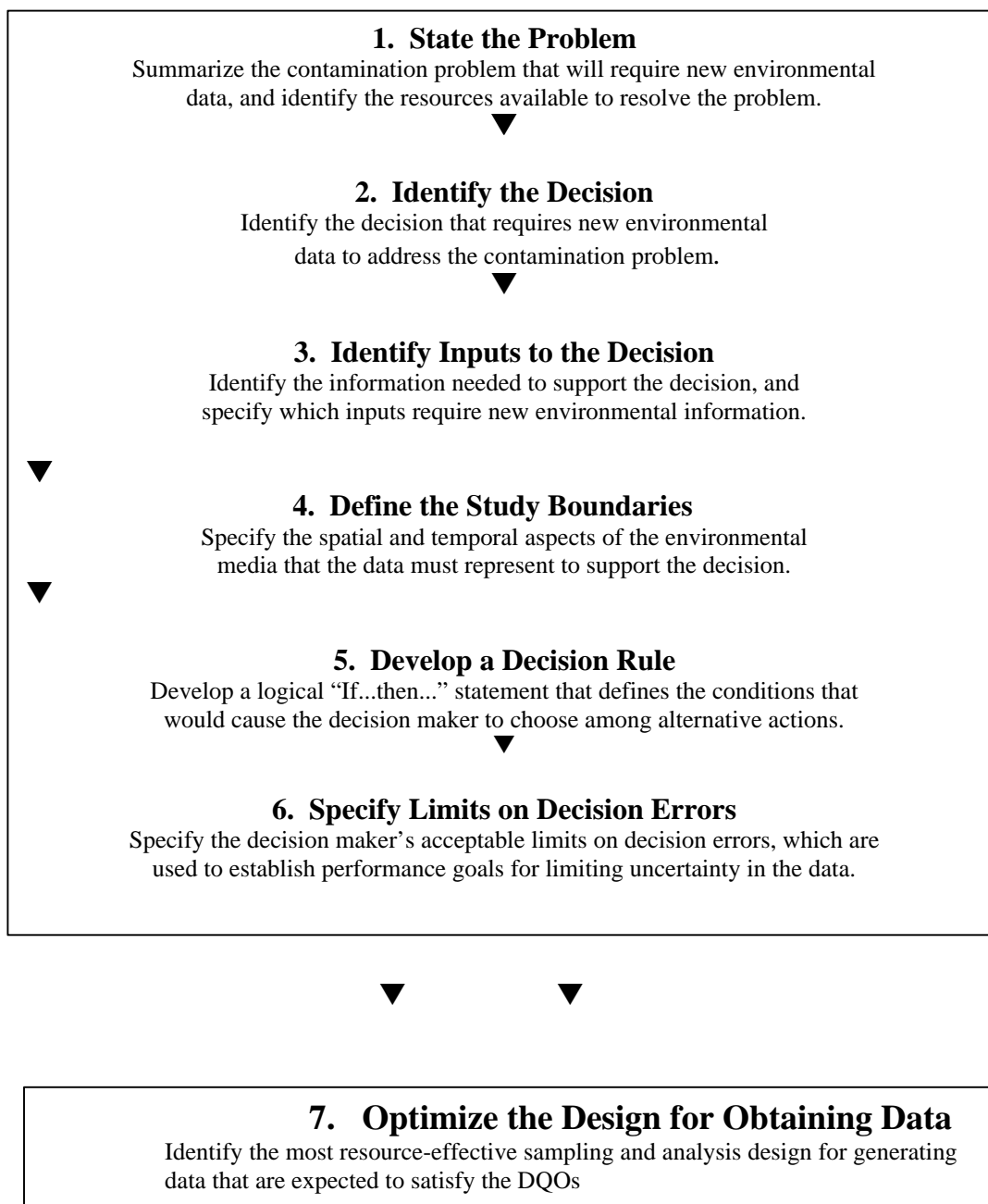
- 1) Clarify the study objective;
- 2) Define the most appropriate type of data to collect;
- 3) Determine the most appropriate conditions from which to collect the data; and
- 4) Specify acceptable levels of decision errors that will be used as the basis for establishing the quantity and quality of data needed to support the decision.

The DQOs are then used to develop a scientific and resource-effective sampling design.³

² Environmental Protection Agency, Office of Emergency and Remedial Response, *Data Quality Objectives Process for Superfund: Interim Final Guidance*, 9355.9-01, EPA540-R-93-071, September 1993, p. 1, NTIS, PB94-963203.

³ Ibid.), 1.

The Data Quality Objective Process



⁴ Environmental Protection Agency, Office of Emergency and Remedial Response, *Data Quality Objectives Process for Superfund: Interim Final Guidance*, 9355.9-01, EPA540-R-93-071, September 1993, p. 2, NTIS, PB94-963203.

In general, a greater amount of information will be required to demonstrate that an area is *not* contaminated (or that concentrations do not pose a health or environmental threat) than to quantitate high levels of contamination for which the facility acknowledges a need for remediation. The DQOs adopted must appropriately reflect the level of information required. Likewise, more attention must be paid to data quality when concentrations of analytes are near to background levels or to analytical detection limits than when contaminant concentrations are significantly above background levels or detection limits. In the example of cleaning up to background, the DQO for the initial site evaluation must be sufficient for determining the vertical and horizontal extent of contamination for the contaminants of concern. At the completion of the project, the DQO for verification sampling must be adequate to demonstrate that background concentrations have been achieved throughout the area of contamination.

Analytical considerations must be evaluated concurrently with factors related to sampling procedures, statistical treatment of data, and (if applicable) the risk assessment process to ensure that the established DQOs can be attained. Facilities must determine the sampling methods, analytical methods, and quality control measures needed to meet the remediation DQO and to generate the documentation (deliverables) required. Facilities are also responsible for notifying the environmental consultants managing their remedial activities and the laboratories analyzing their samples of the required data deliverables *prior* to sampling and analysis activities. This will ensure that appropriate control measures are taken and that documentation of such measures is readily available.

DQOs for Risk Assessment. In the case of risk assessment, very detailed information is generally required to meet the appropriate DQO. This is because in evaluating risk, not only must the extent of contamination⁵ be determined and the concentrations of the contaminants of concern be strictly quantitated, but the *uncertainty* in the measurement of the concentration must also be quantitated. This uncertainty is generated by a variety of factors such as sample characteristics, sampling procedures, analytical procedures, and random variability. In addition, the data must be collected and evaluated while taking into consideration identification of contaminant sources, exposure pathways, exposure criteria, and potential human and environmental receptors.

DQOs for RCRA Closure Projects. In the case of RCRA closure, the initial project objective is to determine whether the area in the vicinity of the unit is contaminated and, if so, to determine the vertical and horizontal extent of contamination and approximate concentrations of contaminants in that area. This, in general, would require less detailed information than would be necessary for a quantitative risk assessment, and the DQOs selected would reflect this.

⁵ Note: Determination of “extent of contamination” for risk assessment might be somewhat different than determination of horizontal and vertical extent has been previously done for RCRA closures. For risk assessment evaluation of soils, determination of extent might involve obtaining two consecutive sample results for which contaminant concentrations meet a health-based “preliminary remediation goal” (PRG) or cleanup level (instead of two consecutive sample results meeting background levels or detection limits). For risk assessment evaluation of ground-water, determining extent might involve sampling to a “point of compliance” (POC), i.e., a geographic surface location within a contaminant plume at which cleanup standards must be met. If the concentration at the POC exceeds the required cleanup standard, sampling would need to continue hydrogeologically down gradient of the POC to the location at which the contaminant concentrations in the plume do meet the required standards.

However, with increasing frequency facilities seeking clean closure are opting to perform risk assessments to demonstrate absence of significant risk. Because the necessity of a quantitative risk assessment often has not been decided upon at the time a closure work plan is initiated, **the IDEM RCRA program is requiring that all sampling and analysis for RCRA closures meet the analytical data deliverables criteria listed in this document.** If this is done and a risk assessment approach is chosen later, sampling and analysis may not have to be repeated in the areas it was already performed for closure purposes, **providing that the extent and sources of contamination, exposure pathways, threshold levels, and other criteria related to risk assessment have been adequately determined.**

DQOs for RCRA Corrective Action Projects. The initial objectives for corrective action at a site consist of determining if Solid Waste Management Units (SWMUs) have released hazardous constituents to the environment and, if so, whether any migration of contamination has occurred (including off site). As with RCRA closure, this involves determining extent of contamination and approximate concentrations of contaminants. Facilities are increasingly making use of risk assessment in the corrective action process, also. **Therefore, the IDEM RCRA program is requiring that all sampling and analysis for RCRA corrective actions meet the analytical data deliverables criteria listed in this document.** If this is done, sampling and analysis may not have to be repeated in the areas it was already performed for RCRA Facility Assessment (RFA) or RCRA Facility Investigation (RFI) purposes, **providing that the extent and sources of contamination, exposure pathways, threshold levels, and other criteria related to risk assessment have been adequately determined.**

DQOs for Enforcement-Related Remediation. DQOs and deliverables for remediation related to enforcement cases generally require information as detailed as that required for risk assessment. This is necessary for legal accountability. It is to the facility's advantage to be able to clearly document that the areas of concern have been adequately characterized: i.e., that the extent of contamination has been determined, *or* that an adequate demonstration has been made that significant levels of contamination are *not* present. If the enforcement action requires remedial action, it is equally important to be able to demonstrate that the remediation has been successfully completed.

GENERAL REQUIREMENTS FOR REMEDIATION-RELATED DELIVERABLES

OLQ Hazardous Waste technical staff must confirm that the analytical data is "valid" (meets DQO criteria) by reviewing the Quality Assurance/Quality Control (QA/QC) data generated during the sampling and analysis procedures and by verifying the analytical and quality control results and calculations from the raw data records. This validation process ensures that technically sound decisions are made which will be protective of human health and the environment. Thus, the QA/QC data and copies of the raw data records necessary for validation are requested in analytical data packages along with final sample results.

OLQ technical staff must also confirm that the site has been sampled in such a manner that contaminants of concern, concentrations of contaminants, and extent of contamination has been adequately determined to meet the project objectives. Therefore, the sampling and analysis plan (SAP), field sheets documenting the sampling activities, and a site map including clearly identified site features (buildings, surface water, roads, boundary, etc.), RCRA units, and sampling points must also be submitted with sampling results.

Sampling QA/QC and Documentation

Meeting the DQOs begins with the sampling process. In addition to the SAP and field sheets, the procedures describing how the sampling operations were actually performed should be provided. Methods from source documents published by the USEPA, American Society for Testing and Materials (ASTM), U.S. Department of the Interior, National Ground-water Association (NGWA), American Petroleum Institute (API), or other recognized organizations with appropriate expertise should be used, if possible. If such standard sampling methods are used, any deviations from the published method should be stated. If sampling methods developed by the facility or facility's consultants are used, copies of such methods should be provided. The procedures for sample collection should be documented with at least the following information:

- Applicability of the procedure,
- Equipment used,
- Measures used to ensure that representative samples were collected,
- Detailed description of procedures that were followed in collecting the samples (including deviations from SAP),
- Field QC measures taken, and
- Field QC samples collected (duplicates, blanks, etc.)

Detailed field QC and deliverables requirements necessary to meet the DQO for analytical data supporting risk assessments are listed later in this document.

Analytical QA/QC and Documentation

The analytical methods used should be recognized standard methods designed for the testing of environmental samples, such as those published by the USEPA and applicable methods published by the ASTM. EPA publication SW-846, *Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, Third Edition* and subsequent updates, is the recommended source for analytical methods. Use of SW-846 methods is only required by regulation in certain instances. "In other situations, SW-846 functions as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in satisfying RCRA-related sampling and analysis requirements."⁶

In remediation projects, the requirement to use SW-846 methods would apply in the following instances: (a) if the remedial action included disposal of hazardous wastes stored or released on the site, or (b) if contaminated media (soil, sediment, ground-water, etc.) generated by the action was slated for disposal or otherwise required characterization as to whether it was a hazardous waste (treatment or storage

⁶ Federal Register, Vol. 60, No. 9, January 13, 1995, p. 3090.

onsite, transportation, etc.).⁷ Specific regulatory citations requiring use of SW-846 methods that could pertain to remedial actions include the following:⁸

1. Section 261.22(a) (1) and (2) -- Evaluation of waste against the corrosivity characteristic;
2. Section 261.24(a) -- Leaching procedure for evaluation of a waste against the toxicity characteristic;
3. Section 261.35(b)(iii)(A) -- Testing rinsates from wood preserving cleaning processes;
4. Sections 264.190(a), 264.314(c), 265.190(a), and 265.314(d) -- Evaluation of a waste to determine if free liquid is a component of the waste;
5. Section 261.112(b)(1) -- Certain analysis in support of exclusion from the definition of a hazardous waste of a residue which was derived from burning hazardous waste in boilers and industrial furnaces;
6. Section 268.32(I) -- Evaluation of a waste to determine if it is a liquid for purposes of certain land disposal prohibitions;
7. Sections 268.40(a), (b) and (f), 268.41(a), and 268.43(a) -- Leaching procedure for evaluation of waste extract to determine compliance with land disposal treatment standards; and
8. Section 268.7(a) -- Leaching procedure for evaluation of a waste to determine if the waste is restricted from land disposal.

For all analytical methods used to generate data that will be submitted to the agency:

- **All quality control measures specified by the method must be followed;** and
- **All quality control criteria and control limits required by the method must be achieved.**

If control criteria cannot be met for technical reasons (such as sample matrix), an explanation must be provided in the case narrative that accompanies the data.

Commonly, the quality control measures and criteria required to validate the data generated by a particular analytical method are included in the body of the method or are attached as tables and appendices. However, to determine *all* applicable QC requirements, it may be necessary to refer to other sections of the reference outside of the determinative method. For example, to comply with all the QC measures required to run SW-846 Method 8260B for volatile organic analysis (VOA), it would be necessary to apply applicable calibration and QC criteria listed in SW-846 Method 8000B (the general instructions applying to all SW-846 gas chromatography methods) and SW-846 Chapter One ("Quality Control", July 1992), which applies to all SW-846 methods, in addition to the measures stated in 8260B. The introductory text of Chapter Four ("Organic Analytes", September 1994), Section 4.1 (Sampling Considerations) would also apply.

Detailed laboratory QC and deliverables requirements necessary to meet DQOs for analytical data supporting risk assessments are listed below. General requirements applicable to all samples are followed by specific requirements by analysis type.

⁷ Requirements regarding contaminated media could change upon promulgation of USEPA's proposed hazardous waste identification rule for contaminated media (HWIR-Media). See "Requirements for Management of Hazardous Contaminated Media; Proposed Rule," *Federal Register*, Vol. 61, No. 83, April 29, 1996, pp. 18779 et seq. Publication of a final HWIR-Media rule is targeted for June 1997.

⁸ *Federal Register*, Vol. 60, No. 9, January 13, 1995, pp. 3089-90.

QUALITY ASSURANCE/QUALITY CONTROL DOCUMENTATION REQUIRED

The following documentation should be submitted with all analytical data reported. This is applicable to all sample matrices and all types of analysis.

Plans Related to Sampling and Analysis:

One copy of all project plans addressing the sampling and analysis activities should be supplied. Examples of applicable documents might include the following:

- Sampling and Analysis Plan (SAP)
- Quality Assurance Program Plan (QAPP)
- Quality Assurance Project Plan (QAPjP)
- Voluntary Remediation Work Plan
- Closure Plan
- RFI Work Plan
- Site Assessment Plan

Sampling Quality Control Data and Information:

- Chain-of-Custody
- Date and time each sample was taken
- Map or diagram indicating sample locations
- Field measurements made (and results)
- Any notable observations (*color, clarity, texture, reaction with preservatives, etc.*)
- Trip blank (or field blank)
- Equipment blank (rinsate blank)
- Identity of field duplicates (a minimum of one duplicate for every 20 or fewer samples)

Laboratory Quality Control Data and Information:

- Completed Chain-of-Custody
- Date and time of receipt at the laboratory
- Condition of samples upon receipt at the laboratory
E.g.: Temperature of cooler (thermometer reading or presence of ice); condition of bottles (cracked? broken? leaking?); condition of samples (pH reading; preserved? air bubbles present?).
- Facility sample identification or number (*e.g., well no.*)
- Laboratory sample numbers corresponding to facility sample identification
- Sample preparation, extraction, cleanup, or digestion method(s) and date(s)
- Analytical method (name, number, and source) and date of analysis
- Final analytical results

- Case narrative:
To include deviations from standard analytical or preparatory procedure(s); quality control problems encountered--whether stemming from system, instrumentation, analyst error, or sample matrix; corrective measures taken; if corrective measures as called for in the method were not taken; results of corrective measures taken; etc.

The laboratory documentation listed on the following pages should be provided according to the analytical method(s) used *in addition* to the **Sampling** and **Laboratory Quality Control Data and Information** listed above. All information pertaining to the method used should be submitted. It may be necessary to explicitly request that the laboratory provide this documentation.

Metals and General Inorganic Analyses

TOTAL AND DISSOLVED METALS by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) or Atomic Absorption Spectroscopy (AA) and GENERAL INORGANIC ANALYSES

- Method/sample quantitation limits
- Instrument detection limits
- Calibration records and results:
 - *Initial calibration:
 - Calibration curve established for each metal
 - **ICP:** A blank plus at least one calibration standard (containing all target analytes) with a minimum of two replicate exposures
 - **AA:** (graphite furnace and flame emission) A blank plus at least three standards
 - **CVAA:** (mercury by cold vapor AA) A blank plus at least five standards
 - **General Inorganic Analysis:** A blank plus at least three standards
Additional requirement for cyanide analyses: a mid-range standard must be distilled and analyzed with results compared to curve for undistilled standards.
 - Correlation coefficient of at least 0.995 for each curve (or calibration is repeated)
 - Concentrations and responses for each standard and blank (numeric)
 - Graphical plot of calibration curve (AA analysis)
 - Date and time of initial calibration
If not the same day as analysis, provide explanation. If this is allowed by analytical method, cite section of method.
 - * Initial and continuing calibration verification (ICV and CCV) (mid-level standard results and % recovery; CCV to be run every ten samples)
- Blank results
 - Initial and continuing calibration blank results
 - Method (preparation) blank results
- Matrix spike (sample number of sample spiked, sample concentration for analyte, concentration of spike added, results and % Recovery)
- Matrix spike duplicate or laboratory duplicate (results and Relative Percent Difference [RPD]; if matrix spike duplicate, also report % Recovery)
- Laboratory control sample (QC standard or lab-fortified blank: results and % Recovery)
- Additional deliverables for ICP analysis:
 - Interference check sample (results and % recovery)
 - Serial dilution results (five-fold analysis)
 - ICP Linear Range
 - Inter-element correction factors

- Additional deliverables for AA analysis if Method of Standard Addition (MSA) is used: data and results for MSA
- **Raw data:** To include instrument numerical printouts, instrument peak printouts (all AA and general inorganic, where applicable), lab worksheets, strip chart recordings, sample preparation records, and record of dilutions.

Organic Analyses

VOLATILE ORGANIC ANALYSIS (VOA) and SEMIVOLATILE ORGANIC ANALYSIS (SVOA) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

- Tuning criteria and results for:
 - VOA: Bromofluorobenzene (BFB) or
 - SVOA: Decafluorotriphenylphosphine (DFTPP)
- Initial calibration data and results:
 - Calibration standards containing all target analytes run at five concentrations
 - Retention time (RT) for each target compound in the calibration standards
 - Response factors (RFs) for each target compound in the calibration standards
 - Average RF for each compound
 - Percent relative standard deviation (RSD) for the RFs for the five concentrations of each calibration standard
 - Date and time of injection
 - Total ion chromatogram
- Initial and Continuing Calibration Verification data and results (beginning of run and every twelve hours):
 - RF for each compound in the 50 ppb standard
 - Percent Difference for RF in 12-hour standard as compared to average RF from initial calibration for each compound
 - Date and time of injection
- Method blank summary sheet with results, including detections
- Detection/quantitation limit for each compound
- Internal standards summary documented by:
 - area of primary peak and respective RT for each standard from the 12-hour standard
 - area of primary peak and respective RT for each standard from each sample
 - upper and lower acceptance limits clearly defined
- Surrogate (System Monitoring Compound) results (concentration of surrogate spikes added, measured concentrations, and % Recoveries of all surrogates) for each sample
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) results (sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound)
- **Raw Data** for each sample, field duplicate, blank, matrix spike, and matrix spike duplicate including:
 - total ion chromatogram (indicating surrogates, internal standards, and target compounds detected)
 - individual mass spectra for target analytes or tentatively identified compounds (TICs, other non-target analytes) detected in each sample and blank (and reference/library search spectra detected analytes or TICs are compared to)
 - quantitation reports (to include identification of internal and surrogate standards, scan number, area, retention time, concentration of target analytes detected, dilution factors, and date and time of injection).

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS and SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY (GC) Using Method-Specified Detectors (FID, PID, HECD, etc.)

- Initial Calibration, data and results documented by:
Either an external standard calibration procedure or an internal standard calibration procedure may be used. Calibration factors (CFs) as defined in SW-846 Method 8000A (July 1992) may be reported in place of response factors.
 - Calibration standards containing all target analytes run at five concentrations
 - Calibration chromatograms
 - Response factors (RFs) or CFs or for each target compound in the calibration standards
 - Average RF (or average CF) for each compound
 - Percent relative standard deviation (%RSD) for the RFs (or CFs) for the five concentrations of each calibration standard
 - Date and time of injection (or introduction by purge-and-trap)
- Retention Time (RT) Summary to include:
 - RT measured for each target compound from three separate injections over a 72-hour period
 - Mean and standard deviations of the three RTs measured (over the 72-hour period)
 - RT window for each target compound (mean \pm three standard deviations)
 - Date and time of injections (or introduction by purge-and-trap)
- Initial and Continuing Calibration Verification (ICV and CCV) documented by:
Note: An instrument blank, a QC reference sample ("check sample"), and a midrange calibration standard should be injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.)
 - Chromatograms for midpoint standard and blank
 - RT for each analyte and (or major peak(s) of each multicomponent analyte, if applicable) in the midrange standard and comparison to daily RT window
 - Percent Difference (%D) between calculated concentration and nominal ("true") concentration of each target analyte in the QC reference sample
 - %D between RF or CF of each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard
- Method of sample introduction (direct injection or purge-and-trap)
- Detection/quantitation limit for each compound
- Method blank summary and chromatograms
- Surrogate recoveries for samples, blanks, and spikes
- Matrix spike/matrix spike duplicate (MS/MSD) analysis (minimum of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix)
OR For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MS/MSD.
- **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, chromatograms and preparatory records.
- Confirmation by GC/MS or on second GC column, if required by determinative method or if interference is suspected. Include results and raw data.

QUALITY ASSURANCE/QUALITY CONTROL INFORMATION FOR ANALYSIS OF PESTICIDES and PCBs by Gas Chromatography (GC) with Electron Capture Detector (ECD) or Electrolytic Conductivity Detector (ELCD or HECD)

- Initial Calibration (Include listing of calibration sequence)
An external standard calibration procedure is preferred, but an internal standard procedure may be substituted. If internal standard procedure is used, report Response Factors (RFs) for each compound at each calibration standard concentration, mean RF, and RF %RSD instead of Calibration Factors (CFs).

*For Single Component Analytes, initial calibration is documented by:

- Five-point calibration preferred; minimum of three-point calibration required.
- Calibration chromatograms must be provided.
- Retention Time (RT) Summary to include:
 - RT measured for each target compound and surrogate at each standard concentration from three-point or five-point calibration
OR RT measured for each target compound from three separate injections over a 72-hour period
 - Mean RT for each target compound and surrogate (mean of three to five RTs from calibration *OR* mean of three RTs measured from injections over a 72-hour period)
 - RT window for each target compound and surrogate
- Calibration Factor (CF) Summary to include:
 - CF calculated for each target compound and surrogate at each standard concentration
 - Mean CF for each target compound and surrogate
 - % Relative Standard Deviation (%RSD) of the CFs at each standard concentration for each compound
- % Breakdown of endrin and % breakdown of DDT
- Date and time of injection

*For multicomponent analytes, initial calibration is documented by:

- Three-point or five-point calibration using mixture of Aroclors 1016 and 1260
- A "one-point calibration" using a midrange standard must be run for all target multicomponent compounds
- Calibration chromatograms must be provided.
- Retention Time (RT) Summary:
 - For Aroclors 1016 and 1260:
 - RT measured for at least one major peak at each standard concentration from the three-point or five point calibration (same peak(s) at each concentration)
OR RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection)
 - Mean RT for the chosen major peak(s)
 - RT window for the chosen major peak(s)

- Initial and Continuing Calibration Verification (ICV and CCV) documented by:

Note: An instrument blank, a QC reference sample (“check sample”), and a midrange calibration standard is injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.) For PCBs only Aroclors 1016 and 1260 need be injected unless there are specific known target PCBs at the site. If so, all targeted PCBs should be injected.

 - Chromatograms for midpoint standard and blank
 - Absolute RT for each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard (and comparison to RT window established at calibration)
 - Percent Difference (%D) between calculated concentration and nominal (“true”) concentration of each target analyte in the QC reference sample
 - %D between RF or CF of each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard
 - For multicomponent analytes run at midrange concentration only:
 - RT measured for three to five major peaks from “one-point calibration” run
OR RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection)
 - Mean RT for the chosen major peak(s)
 - RT window for the chosen major peak(s)
 - Calibration Factor (CF) Summary to include:
 - CF calculated for each target compound (total area of all peaks used for quantitation) at each standard concentration (or from each of three injections)
OR CF calculated for three to five major peaks of each target compound from calibration run of midpoint standard
 - Mean CF for each target compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only)
 - % Relative Standard Deviation (%RSD) of the CFs for each compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only)
 - % Breakdown of endrin and % breakdown of DDT
 - Date and time of injection
- Method blank summary and chromatograms
- Detection/quantitation limit for each compound (in each sample)
- Surrogate recoveries for samples, blanks, and spikes
- Matrix spike/matrix spike duplicate (MS/MSD) analysis (minimum of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix)
OR For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MS/MSD.
- **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, preparatory records, and chromatograms
- Confirmation of detection **required:** on second GC column OR by GC/MS

- Chromatograms for samples, blanks, spikes, and standards for confirmation run on second column must be provided.
- If confirmation is done by Gas Chromatography/Mass Spectroscopy (GC/MS), the following information (relevant to GC/MS analysis) should also be provided:
 - Tuning criteria and results (instrument performance check)
 - Calibration records (including total ion chromatogram)
 - Chromatograms for samples and method blank
 - QC reference sample for detected compounds
 - Mass spectra for samples, QC reference sample, and blank, including reference spectra for detected compounds

ADDITIONAL SOURCES OF ANALYTICAL METHODS FOR ENVIRONMENTAL SAMPLES

The following references are examples of methods manuals in addition to SW-846 that would be acceptable for use in remediation project sample analysis:

- "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, March 1979 (Revised March 1984)
- "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R-93/100
- "Methods for the Determination of Organic Compounds in Drinking Water," EPA 600/4-88-039 (1991 Update), U.S. Environmental Protection Agency, July 1991
- "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA 600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.
- *Standard Methods for the Examination of Water and Wastewater*, 19th Edition, 1995
- "USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis: ILM03.0"
- "USEPA Contract Laboratory Program Statement of Work for Organic Analysis: OLM03.1"

REFERENCES

“Analytical Data Deliverable Requirements for RCRA Permits: A Guidance Document,” IDEM OSHWM Technical Waste Assessment Section, (Draft) March 1995.

Federal Register, Vol. 60, No. 9, January 13, 1995, pp. 3089 et seq.

Federal Register, Vol. 61, No. 83, April 29, 1996, pp. 18779 et seq.

U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, *Data Quality Objectives Process for Superfund: Interim Final Guidance*, EPA-540-R-93-071, PB94-963203, September 1993.

U.S. Environmental Protection Agency Region 5, Waste Management Division, Office of RCRA, “Ecological Risk Assessment Guidance for RCRA Corrective Action: Region 5: Interim Draft.” Chicago: USEPA Region 5, October 1994.

U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Toxics Integration Branch, *Risk Assessment Guidance for Superfund, Volume I, Human Health Evaluation Manual (Part A), Interim Final*, EPA/540/1-89/002, December 1989.

U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Toxics Integration Branch, *Risk Assessment Guidance for Superfund, Volume I, Human Health Evaluation Manual (Part B), Development of Risk-based Preliminary Remediation Goals*), OSWER Directive 9285.7-01B, December 1991.

U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Toxics Integration Branch, *Risk Assessment Guidance for Superfund, Volume II, Environmental Evaluation Manual: Interim Final*, EPA/540/1-89/001, March 1989.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, *Test Methods for Evaluating Solid Wastes, Physical/ Chemical Methods*, USEPA Publication SW-846:

- Third Edition, November 1986
- Third Edition Final Update I, July 1992
- Third Edition Final Updates II and IIA, September 1994
- Third Edition Final Update IIB, January 1995.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, Publication 9240.1-05, EPA-540/R-94/012, PB94-963501, February 1993.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, *USEPA Contract Laboratory Program Statement of Work for Organic Analysis: Multi-Media, Multi-Concentration, Revision OLM03.1*, EPA-540/R-94/073, PB95-963503, August 1994.e repeated in the areas it was already performed for closure purposes, **providing that the extent and sources of contamination, exposure pathways, threshold levels, and other criteria related to risk assessment have been adequately determined.**